IS 10300 (1982): Captafol, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard
SPECIFICATION FOR CAPTAFOL, TECHNICAL

UDC 632·952 CAPTAFOL

INDIAN STANDARDS INSTITUTION
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NEW DELHI 110002

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SPECIFICATION FOR CAPTAFOL, TECHNICAL

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Indian Standard

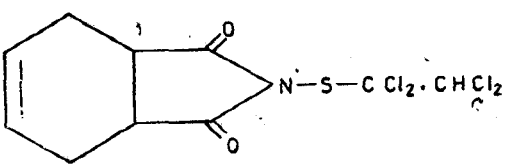
SPECIFICATION FOR CAPTAFOL, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 September 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Captafol, technical is used in fungicidal formulations.

0.3 Captafol is the common name accepted by the International Organization for Standardization (ISO) for N — (1,1,2,2 — tetrachloroethyl) thio — 4 — cyclohexene — 1,2 — dicarboxymide. The empirical and structural formulae and molecular mass are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{10}H_9Cl_4NO_2S$		349

0.4 In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the round off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for captafol, technical.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of light to yellow powder free from extraneous matter.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR CAPTAFOL, TECHNICAL

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Appendix	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Captafol content, percent by mass, *Min*	95·0	A	—
ii)	Moisture content, percent by mass, *Max*	2·0	—	4
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	2·0	—	11·3

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in mild steel or tinplate or fibreboard containers or double hessian jute bags (*see* IS : 8115-1976) or DW tarpaulin laminated jute bags (*see* IS : 8117-1976) or HDPE woven sacks (*see* IS : 8069-1976). The containers shall also comply with the general requirements as specified in **2** of IS : 8190 (Part I)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information as required under the *Insecticides Act and Rules*:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Captafol content, percent (m/m);
f) Net mass of the contents; and
g) A cautionary notice as worded in *Insecticides Act and Rules*.

*Requirements for packing of pesticides: Part I Solid pesticides (*first revision*).

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the 'Indian Standard methods of sampling for pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the parties concerned.

5. TESTS

5.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distillied water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (*i*)]

DETERMINATION OF CAPTAFOL CONTENT

A-0. PRINCIPLE

A-0.1 Captafol is separated from chlorine containing impurities by silicic acid column chromatography. Captafol fraction from the column is quantitatively decomposed to chloride ion by sodium biphenyl reagent. The liberated chloride is then titrated via Volhard's procedure.

*Specification for water for general laboratory use (*second revision*).

A-1. REAGENTS

A-1.1 Silicic Acid — chromatographic grade.

A-1.2 Benzene — analytical reagent grade.

A-1.3 Sand — washed.

A-1.4 Ether-Benzene Mixture — 1 : 1 (*v*/*v*) (anhydrous reagent grade ether).

A-1.5 Benzene-Hexane Mixture — 7 : 3 (*v*/*v*).

A-1.6 Acetone — analytical reagent grade.

A-1.7 Nitric Acid — 6 percent (*v*/*v*).

A-1.8 Sodium Biphenyl Reagent — 30 percent (*m*/*m*).

A-1.9 Phenolphthalein Indicator Solution — 1 percent (*m*/*v*) in absolute alcohol.

A-1.10 Dilute Nitric Acid — 1 : 1 (*v*/*v*).

A-1.11 Standard Silver Nitrate Solution — 0·1 N.

A-1.12 Hydrazine Sulphate — Crystals.

A-1.13 Hydrogen Peroxide — 30 percent (*v*/*v*).

A-1.14 Standard Potassium Thiocyanate Solution — 0·1 N.

A-1.15 Ferric Alum Indicator — Saturated, aqueous, freshly prepared.

A-1.16 Toluene — Chloride free.

A-1.17 Sodium Hydroxide Solution — 30 percent (*m*/*m*).

A-1.18 Nitrobenzene

A-2. APPARATUS

A-2.1 Chromatographic Column — 25 cm long and 25 mm internal diameter with teflon stopcock outlet and the bottom of the column fitted with a fritted glass disc that supports the absorbent and 100-ml reservoir at the top.

A-3. PROCEDURE

A-3.1 Preparation of Column — The silicic acid should be hydrated to the extent of 10 percent. This can be accomplished by first drying at 150°C for several hours and finally shaking for several hours with 10 percent by mass of distilled water (*see* Note). Shake 20 g of the hydrated silicic acid with 50 ml of benzene. Stir the mixture with a glass rod until all the lumps have been broken up and no entrapped air bubbles remain. Drain the

liquid, rinsing down the walls of the column with more benzene. Do not allow the liquid to drop below the top of the silicic acid column. Put 1 to 2 cm layer of the washed sand on top of the column to prevent disturbance of the silicic acid when the solvents are added.

NOTE — If the silicic acid is too dry, breakdown of captafol may occur on the column. If too wet, absorption of captafol on the column may not be completed.

A-3.2 Accurately weigh about 0·25 g of captafol, technical and dissolve in about 25 ml of benzene. Pour the solution on to the column, rinsing with a small quantity of benzene. Using air pressure, force the liquid into the column. Again do not allow the liquid level to drop below the top of the column. Rinse the column with four 25 ml portions of benzene-hexane mixture (*see* **A-1.5**). Rinse off the tip of the column with a small amount of acetone. Discard the benzene-hexane washes and the acetone rinse.

A-3.3 Elute captafol from the column with four 25 ml portions of ether-benzene mixture (*see* **A-1.4**) into a 400 ml beaker. Push the last amount of eluent completely through the column, allowing the column to go dry. Rinse off the tip of the column, which may have crystals of captafol on the outside, with a small amount of acetone, collecting this rinse with the ether-benzene eluate.

A-3.4 Add a boiling chip to the eluate and gently boil off most of the solvent leaving approximately 5-10 ml. Transfer quantitatively into a 125 ml separating funnel using 25-30 ml of toluene. Cautiously add 10-14 ml of sodium biphenyl reagent, mix by swirling and let stand for 5 minutes. If solution is not dark green, add additional 10-14 ml reagent and keep for 15 minutes.

A-3.5 Destroy excess reagent by dropwise addition of water, shaking frequently between additions until green colour is completely removed. Then slowly add 25 ml dilute nitric acid (6 percent *v/v*) with intermittent swirling. Stopper the separator and mix with gentle rocking motion, venting occasionally. Avoid vigorous shaking during the first extraction. Let separate, rinse the stopper and walls of separating funnel with water, and drain the aqueous phase into a 250 ml erlenmeyer flask. Re-extract the mixture with two 25 ml portions of dilute nitric acid (6 percent *v/v*) by shaking vigorously. Add aqueous extracts into the erlenmeyer flask.

A-3.6 Add 30 percent sodium hydroxide to the acid solution in the erlenmeyer flask carefully until alkaline to the phenolphthalein, and add 1 ml excess. Add 5 ml of 30 percent hydrogen peroxide solution, heat to boiling on hot plate, and boil for approximately 10 minutes. Let it cool slightly, cautiously add 5 ml more of 30 percent hydrogen peroxide (H_2O_2) and boil again for about 10 minutes. Cool and add a small flake (about 0·05 g) of hydrazine sulphate to remove the last traces of hydrogen peroxide.

A-3.7 Neutralise the solution with nitric acid (*see* **A-1.10**) using phenolphthalein. Add 10 ml in excess. A known excess of 0·1 N silver

nitrate solution is added accurately and back titrated with standard potassium thiocyanate solution using ferric alum as indicator and nitrobenzene to coagulate the silver chloride formed. Run a blank with the product to make a correction for the inorganic chloride.

A-3.8 Calculation

$$\text{Captafol content, percent by mass} = \frac{8{\cdot}73 \times (B - A) \times N}{M}$$

where

B = volume, in ml, of potassium thiocyanate solution required for the blank determination;

A = volume, in ml, of potassium thiocyanate solution required for sample;

M = mass, in g, of sample taken for the test (*see* **A-3.2**); and

N = normality of potassium thiocyanate solution.

(*Continued from page* 2)

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